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OF A VARIETY OF MATERIALS HAVING STABLE EMITTANCE CHARACTERISTICS

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A METHOD FOR MEASURING THE SPECTRAL NORMAL EMITTANCE IN AIR OF A VARIETY OF MATERIALS HAVING STABLE EMITTANCE CHARACTERISTICS

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ABSTRACT

A method and apparatus is described for the measurement of spectral normal emittance in air of a variety of materials. The system permits measurements to be performed over a wavelength region of 1.0 through 15.0 microns and over a temperature range of 600° F to 1,800° F with an accuracy of 5.0 percent. The advantages of this system are described. Results obtained by this system are compared with results reported by another observer using a different technique.

INTRODUCTION

In order that the proper selection of materials for the design of space radiators, heat rejection systems, and power plants for use in space can be made, accurate heat-transfer calculations must be performed. Since the spectral emittance of these materials plays an extremely important part in these calculations, a program has been initiated at the Langley Research Center of NASA for the investigation of these properties. One phase of the spectral emittance program is the measurement of spectral normal emittance in air of a variety of materials with stable emittance properties. These materials include ceramics, cermets, stably oxidized metals, and coatings on metallic substrates.

In efforts to provide an improved system for obtaining these measurements, a technique together with necessary apparatus was developed which has proved to be relatively simple, fast, and reliable. The apparatus utilized in this system consisted of a modified commercially available recording spectrophotometer and a blackbody furnace similar to that used by McMahon in his investigation of glasses in reference 1. This system permits measurements of spectral normal emittance to be made over a temperature range from 600° F to 1,800° F. Since the primary use of the data obtained by this system will be for heat-transfer calculations, the near infrared region from 1.0 through 15.0 microns was chosen. This region, according to Planck's spectral energy distribution law, contains a minimum of 90 percent of the energy in the 600° F to 1,800° F temperature range.

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The description of this system and the procedure used to obtain these measurements is the subject of this paper.

APPARATUS

The apparatus under consideration can be divided into two main parts. A spectrophotometer with its detector and recorder is one part. A furnace, which contains the test specimen and a blackbody cavity, and an associated temperature monitoring system and power supply comprise the second part. These two parts of the apparatus are described in detail in the following sections.

Spectrophotometer and Associated Apparatus

A commercially available spectrophotometer is used in this investigation. A minor modification was made to the optical system to allow the use of an external source. These modifications together with the original optical path are shown schematically in figure 1. Mirrors M_1 and M_2 as well as the internal source of the standard instrument, were removed. Mirrors M_3, a 6-inch focal-length parabolic mirror, and M_4, a plane-surface mirror, were positioned to transmit radiation from the external source and to focus this radiation at the same point as the original instrument optics. A new cover for this portion of the instrument was designed with an aperture and water-cooled shutter so that the radiation from the external source could be prevented from passing into the instrument when so desired.

Since this spectrophotometer has several interchangeable prisms and detectors available for use in various regions of the spectrum, the sodium chloride prism with the high-speed evacuated thermocouple detector with a potassium bromide window were chosen to cover this spectral region, from 1.0 through 15.0 microns.

Furnace and Associated Apparatus

The furnace, shown in figure 2, is designed similarly to that used by McMahon (ref. 1) in his investigations of glasses. It is constructed essentially of three cubes. The inner cube, called the liner, serves as the blackbody cavity. It is composed of heavily oxidized Inconel approximately 1/16 inch thick and 6 inches on each side. This liner fits inside a cube of silicon carbide which serves as the furnace core. The silicon carbide is wound with 17-gage (AWG) Nichrome V resistance wire that serves as the heating element. The core is well insulated by an outer cube of high-temperature refractory firebrick. All these cubes are fabricated in two halves to facilitate specimen placement. The lower half of the furnace contains a blackened, tapered, water-cooled viewport, as well as provisions for rotating the test specimen within 1/16 inch of the Inconel liner by an external 15-rpm motor.

Since the furnace is divided into two halves and the Nichrome V windings are separated, the power is supplied and controlled by two continuously variable autotransformers.

In operation 30-gage (AWG) chromel alumel thermocouples are spotwelded to the outer surface of the Inconel liner. These thermocouples are constantly monitored to adjust and hold the temperature of the cavity (and thus the temperature of the test specimen) at the desired value.

TEST PROCEDURES

Specimens used for this investigation were cut from 1/8-inch stock in the form of a 4-inch-diameter semicircle. These specimens were positioned on the rotating shaft in the furnace normal to the axis of the viewport and passing within 1/16 inch of the viewport for each cycle of rotation. By manual control of the autotransformers, the furnace cavity is brought to equilibrium at the desired temperature, and emittance measurements are begun.

An example of a typical recorder trace showing the measuring technique is shown in figure 3 for a particular temperature and wavelength. The recorder zero V_O is displaced up scale to allow the complete monitoring of any change in this setting. The monochromator is then set at the desired wavelength and the water-cooled shutter is opened. The radiant flux intensities from the blackbody reference V_B and from the test specimen V_S are then recorded and the water-cooled shutter is closed. This procedure is repeated at 0.5-micron intervals from 1.0 through 15.0 microns. The monochromator slits are held constant and the instrument amplifier gain is adjusted at each reading in order that a maximum recorder deflection can be obtained. The large recorder deflection minimizes the error in calculating emittance values from recorder charts. The calculation of the spectral normal emittance $E_{n\lambda}$ is then obtained by the ratio:

$$E_{n\lambda} = \frac{V_S - V_O}{V_B - V_O}$$

CALIBRATION

Spectrophotometer

Since several prisms are interchangeable for this spectrophotometer, the wavelength indicator is divided in arbitrary units. Each prism used in this instrument must, therefore, be calibrated through the wavelength region covered by this prism. Since the infrared region from 1.0 through 15.0 microns is of interest in this study, the sodium chloride prism was chosen. This prism was then calibrated through this region by the use of absorption and emission

spectra of known compounds and elements and a calibration curve of wavelength indicator number as a function of wavelength was constructed.

Furnace

In order to determine whether any possible temperature differential exists between a test specimen and the cavity, an Inconel specimen with three thermocouples attached, as shown in figure 4(a), was placed in the furnace out of view of the port. The temperature of the cavity was measured by thermocouples attached at eight points on the Inconel cavity. The placement of these eight thermocouples is shown in figure 4(b).

The temperatures of the specimen and of the cavity were measured over the temperature range from 600° F to 1,800° F and were found to be in close agreement as shown in figure 5. Although these temperatures are averages of all thermocouples on the specimen or cavity, the actual differences are less than 1.0 percent. The specimen temperature was lower than the cavity temperature over the entire temperature range, but the maximum deviation was only 0.87 percent of the absolute temperature. These measurements indicate that the assumption of thermal equilibrium between specimen and cavity is valid for the non-measuring portion of the specimen cycle. Thus, the measurement of cavity temperature may be taken also as specimen temperature with a possible emittance error of less than 3.50 percent.

When the specimen is rotated in front of the viewing port it radiates to the cooler surroundings and obviously must cool. The rate of cooling is determined by many factors, one of which is the emittance of the specimen. To determine the effects of this cooling on emittance measurements the following procedure was used: An Inconel specimen was placed in the furnace, out of view of the port, and heated to 1,800° F. After the furnace temperature was stabilized, measurements of radiant flux intensity were obtained by use of a total radiation bolometer. With the specimen out of view of the port the bolometer was focused into the cavity, and a measurement of the blackbody flux intensity was recorded. The specimen was then rotated past the viewport, and a measurement of the specimen flux intensity was recorded. This procedure of obtaining measurements for the blackbody and specimen alternately was continued for 12 cycles. The results are shown in figure 6 where

f_S radiometer output for specimen, rms volts

f_B radiometer output for blackbody, rms volts

From a comparison of the ratio of the radiant flux intensities of specimen and blackbody, an indication of the cooling effect is obtained. The specimen emittance measured for the 1/2 cycle (the first viewing cycle) was 0.95, and that measured for the 3/2 cycle was 0.94. This difference is about 1.00 percent, which indicates that the specimen cooling from rotating past the viewport is negligible for most materials.

The reproducibility of this system as measured during these tests is within 0.5 percent.

The total error possible in considering all these factors cannot exceed 5.00 percent and is probably much less.

ADVANTAGES OF SYSTEM

This system for the measurement of normal spectral emittance in air of materials with stable emittance properties at moderately high temperatures has several distinct advantages over many other systems used in these studies.

One advantage is the elimination of attached thermocouples on the surface of the specimen or use of other temperature measuring devices, such as optical pyrometers, to obtain the temperature of the test specimen. The temperature of the cavity is measured by thermocouples spotwelded to the outer surface of the cavity and the specimen temperature can be maintained to within 1.00 percent of the cavity temperature, which makes the system practical for use with a large variety of materials. These include ceramics, cermets, thick coatings on metallic substrates, and other materials to which it is difficult or impossible to attach a thermocouple.

Methods utilizing a dual-beam spectrophotometer for these measurements are complicated by the cost and time involved in equalizing the path lengths and absorption of mirrors in each beam. Since a separated specimen and blackbody reference is used in that type of operation, a problem of maintaining them at equal temperatures is present. On the other hand, methods favoring single-beam operation and separated reference and specimen are confronted with the same problem of temperature control or the problem of absorbing gases in the optical path.

The use of the blackbody-cavity method requires only one path, so that the problem of equalizing the beams, as in dual-beam operation is avoided. The blackbody reference and test specimen are kept at approximately equal temperatures, with differences of less than 1.00 percent, by control of the furnace temperature alone; thus the need for elaborate temperature regulating devices is eliminated. The reference and test specimen are viewed within 2 seconds, so that the effects of changes in composition and quantity of absorbing gases in the optical path are canceled out.

TYPICAL RESULTS

Measurements of normal spectral emittance using this method have been performed on several high-temperature materials including boron nitride, Inconel, Inconel-X, alumina, and zirconia as well as several other refractory metals. The apparatus is currently being used to study flame sprayed ceramics on refractory metals.

Figures 7, 8, and 9 present comparisons of the results obtained by this system and those obtained by Henry H. Blau, Jr. and associates using a different technique, as reported in reference 2. The NASA data shown in figure 7 were taken from reference 3.

The measurements reported by Mr. Blau and associates were made on test specimens heated in air at temperatures from 600° C to 1,000° C and over a wavelength region from 2.0 to 15.0 microns, with a reported accuracy of ±4 percent. The procedures used differed from those described in this paper in that temperatures of both specimen and blackbody were measured by embedded thermocouples and values of radiant intensity from the specimen and blackbody reference were measured independently. Although the specimen and blackbody heat source varied from those used in this investigation, almost all other apparatus and techniques were very similar to those reported in this paper.

One main problem discussed in reference 2 is that of determining the temperature lag of the thermocouples used to obtain the temperature of the blackbody and specimen.

Another problem, although not discussed in reference 2, is the control of absorbing gases in the optical path due to the time interval necessary between measurements of specimen and blackbody radiation. The temperature and humidity of the room were closely controlled so that the errors caused by these absorbed gases would be extremely small.

As illustrated in figures 7, 8, and 9, agreement in the results obtained by the two systems is extremely close, and the error in each system is approximately the same. Therefore, the choice between these two systems depends on such other factors as the ease of specimen preparation and of operation of the apparatus.

CONCLUDING REMARKS

A system has been developed for the measurement of spectral normal emittance in air of materials with stable emittance properties. The variety of materials which can be tested, the simple method of specimen and blackbody temperature control, and the elimination of problems associated with absorbing gases in the optical path are some of the advantages of this system over others now in use.

Both consideration of possible errors and comparison of results with those of a prior investigator indicate that the results are accurate and reliable.

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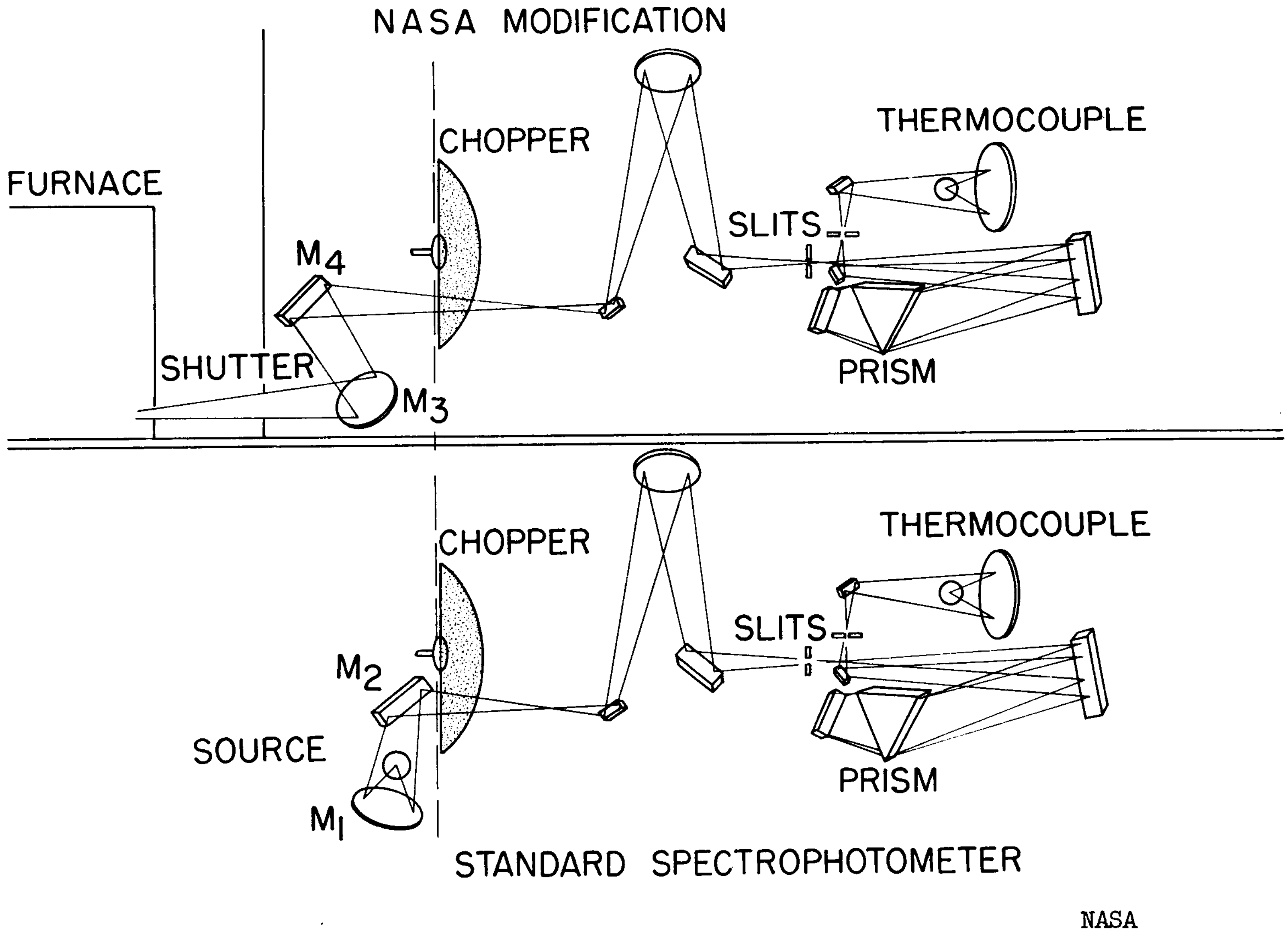

Figure 1.- Optical schematic of spectrophotometer.

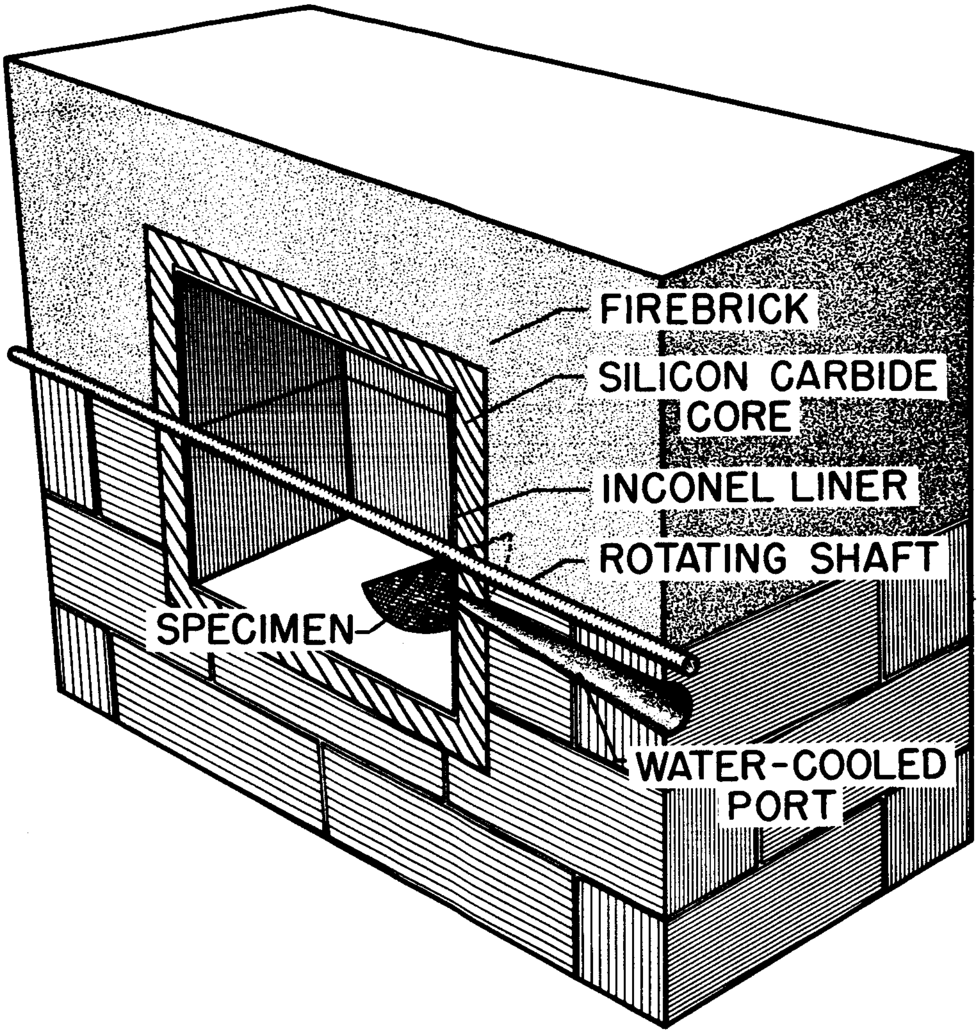

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Figure 2.- Cross section of blackbody furnace.

$$\epsilon_{n\lambda} = \frac{V_S - V_O}{V_B - V_O}$$

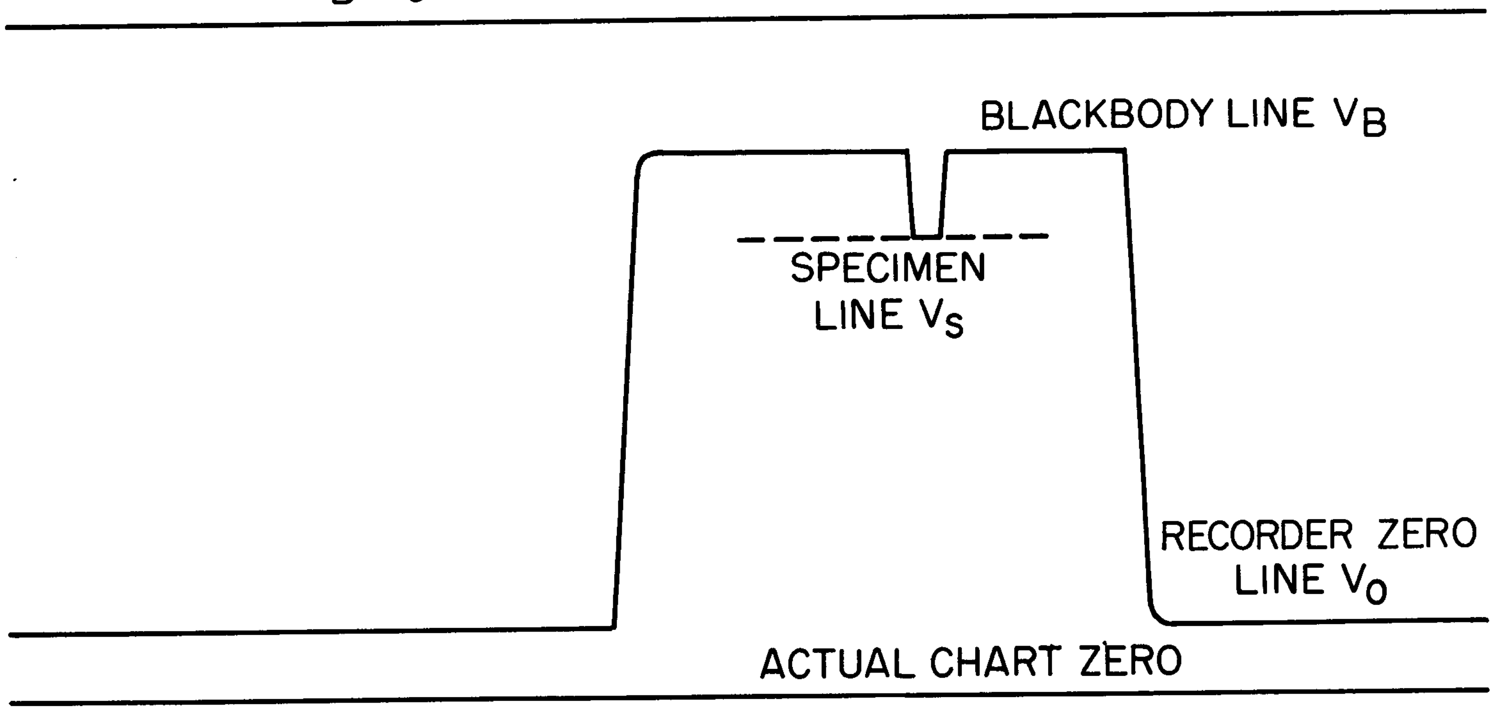

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Figure 3.- Typical data trace.

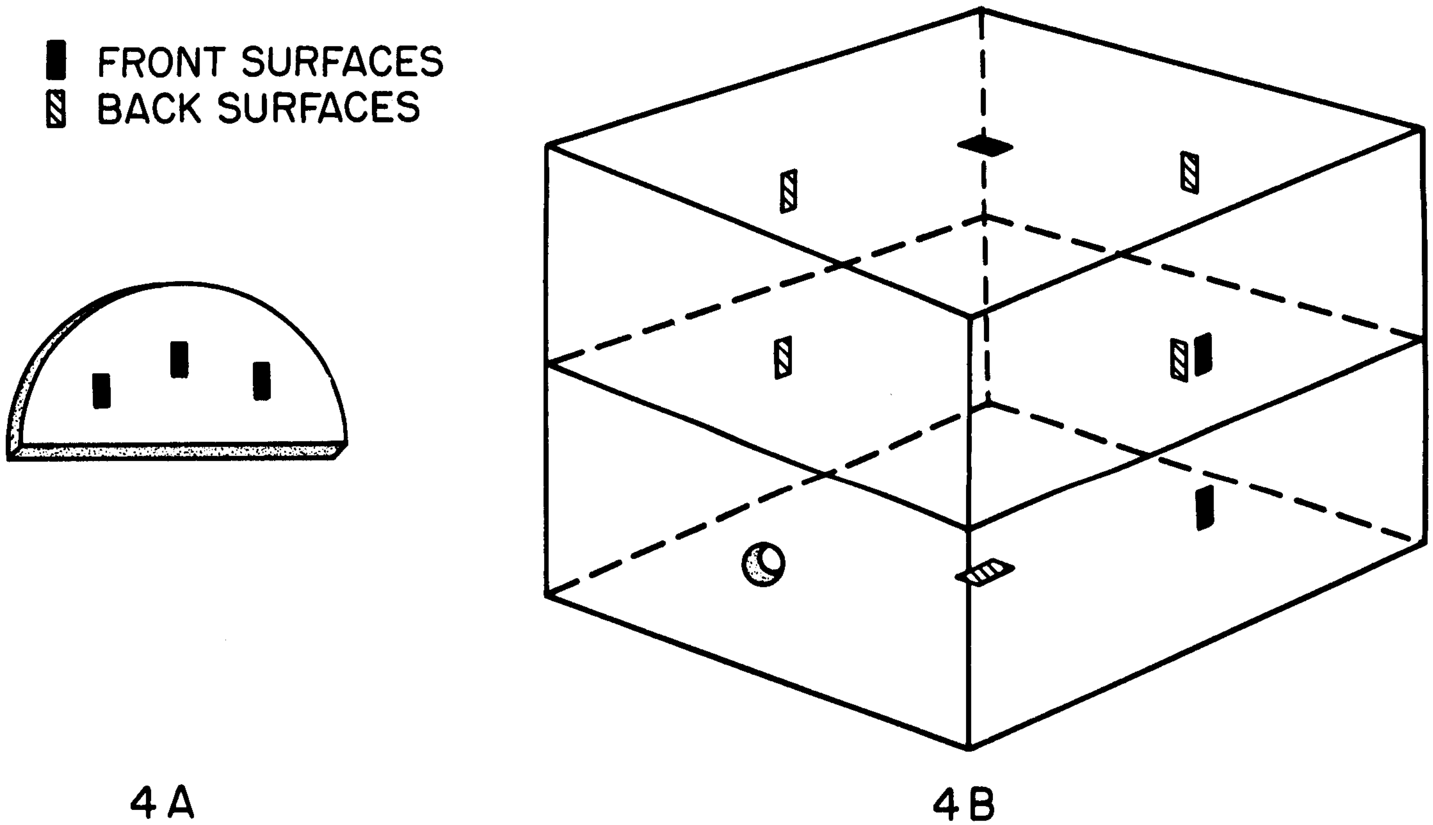

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Figure 4.- Thermocouple placement on specimen and cavity.

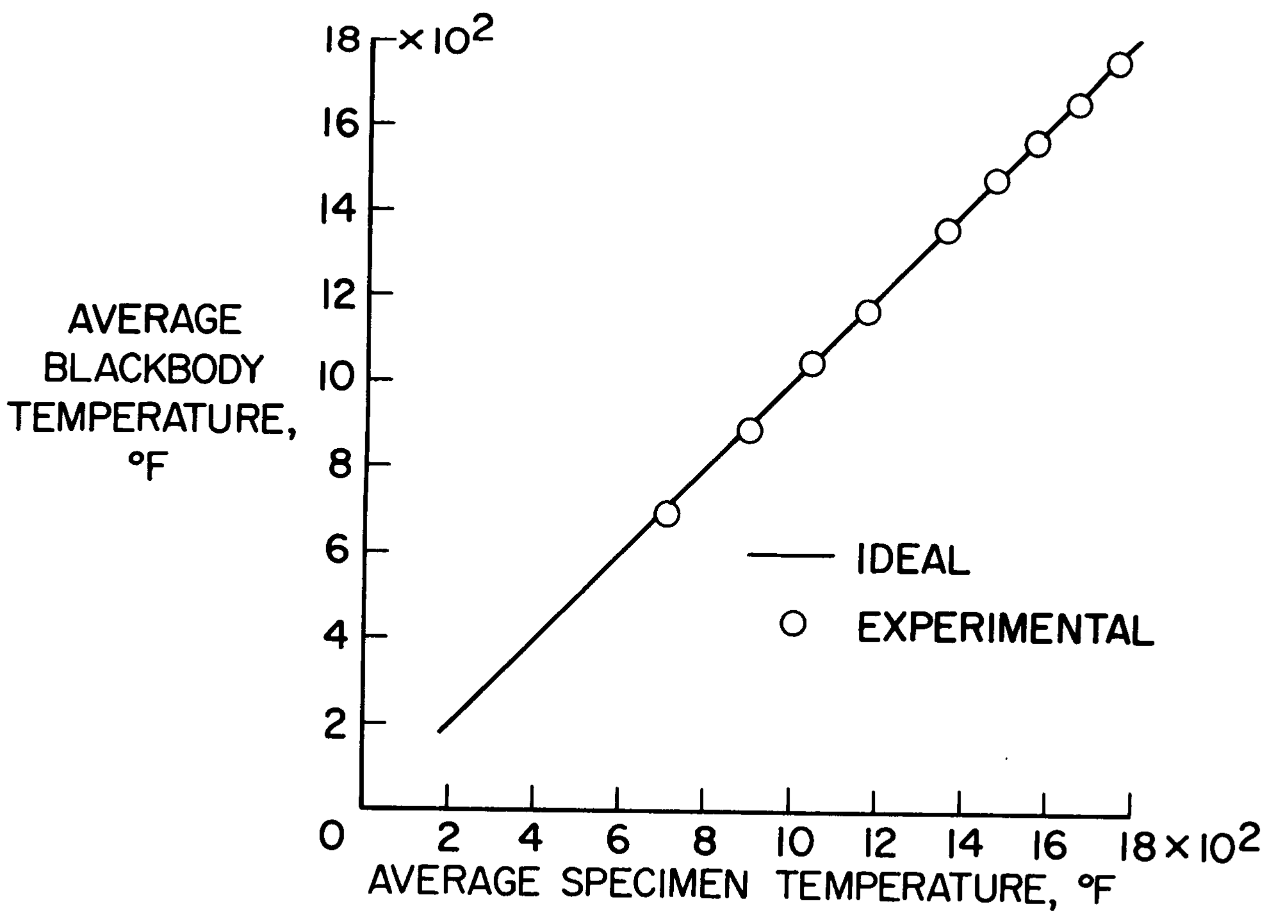

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Figure 5.- Deviation of specimen temperature from blackbody temperature.

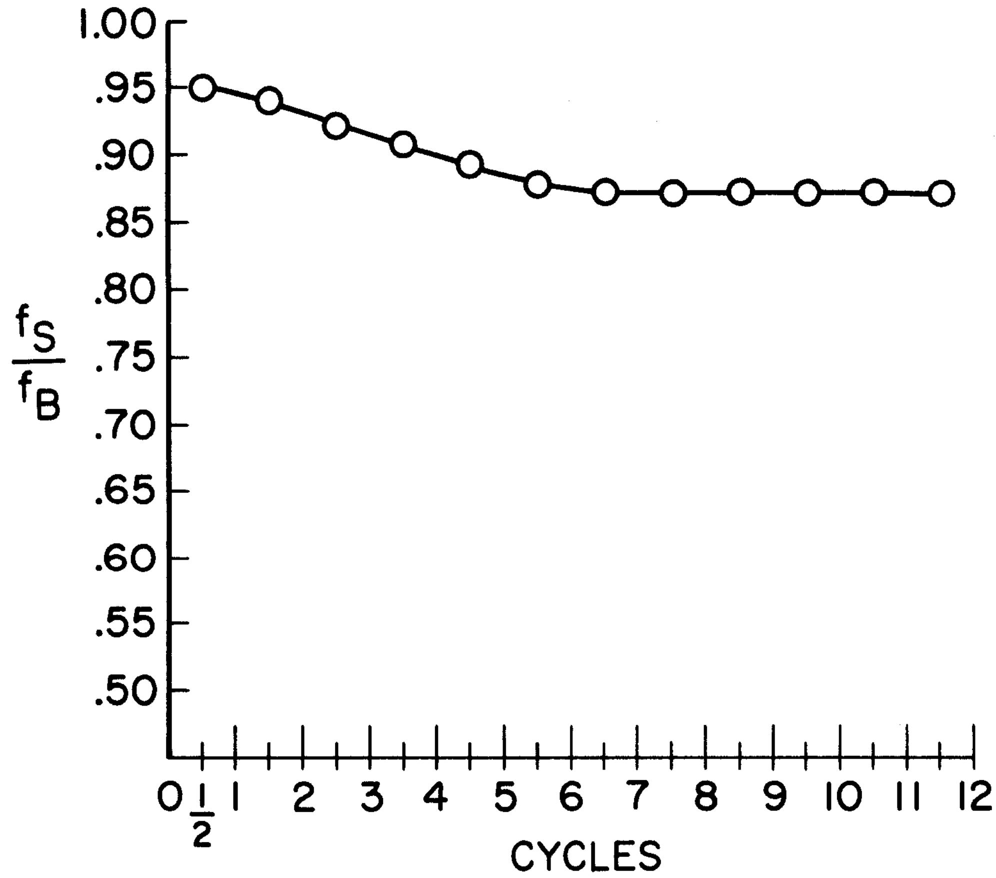

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Figure 6.- Decrease of radiant intensity ratio for specimen of grit blasted Inconel X at a furnace temperature of 1,800° F.

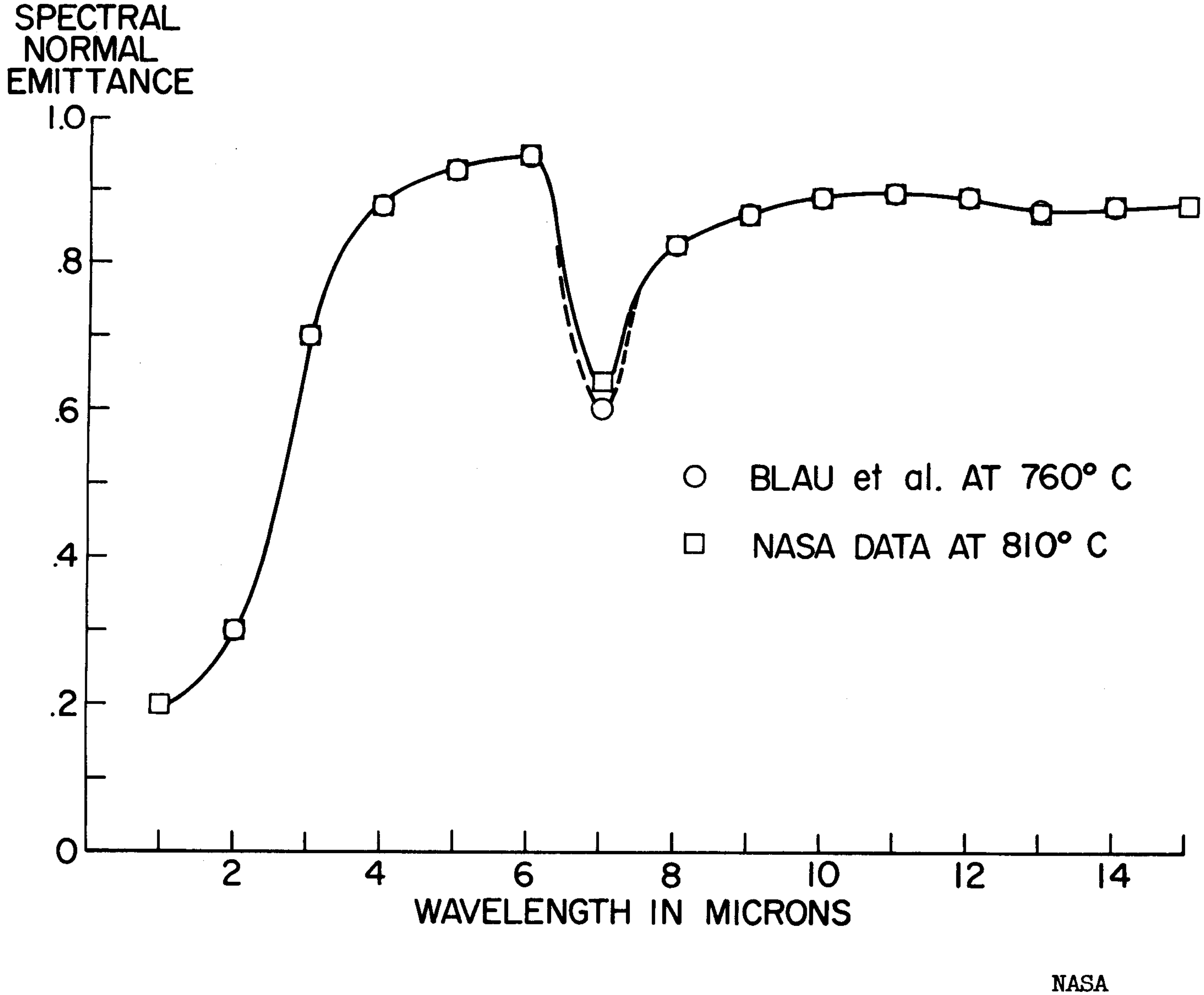

Figure 7.- Boron nitride.

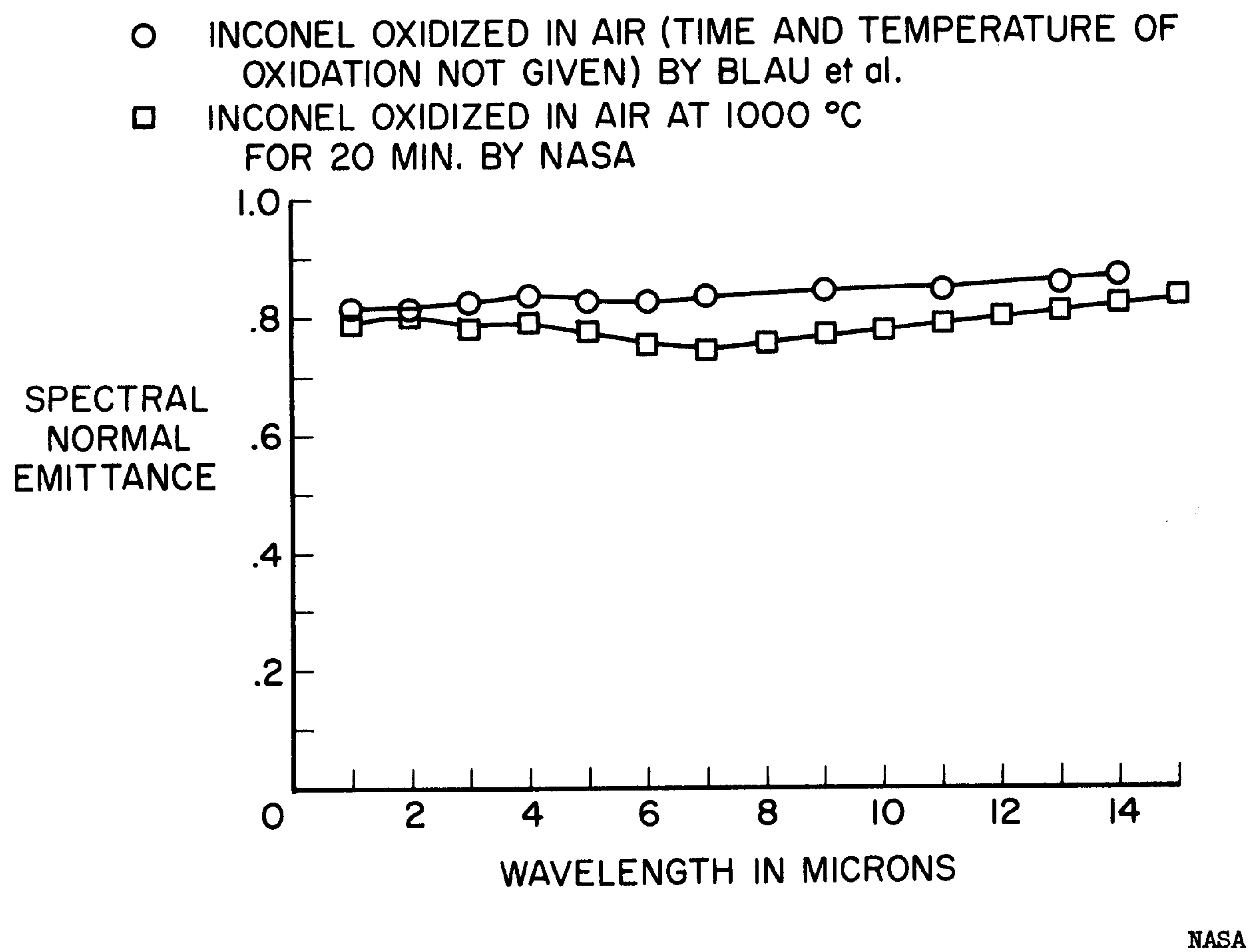

Figure 8.- Oxidized Inconel at 1,000° C.

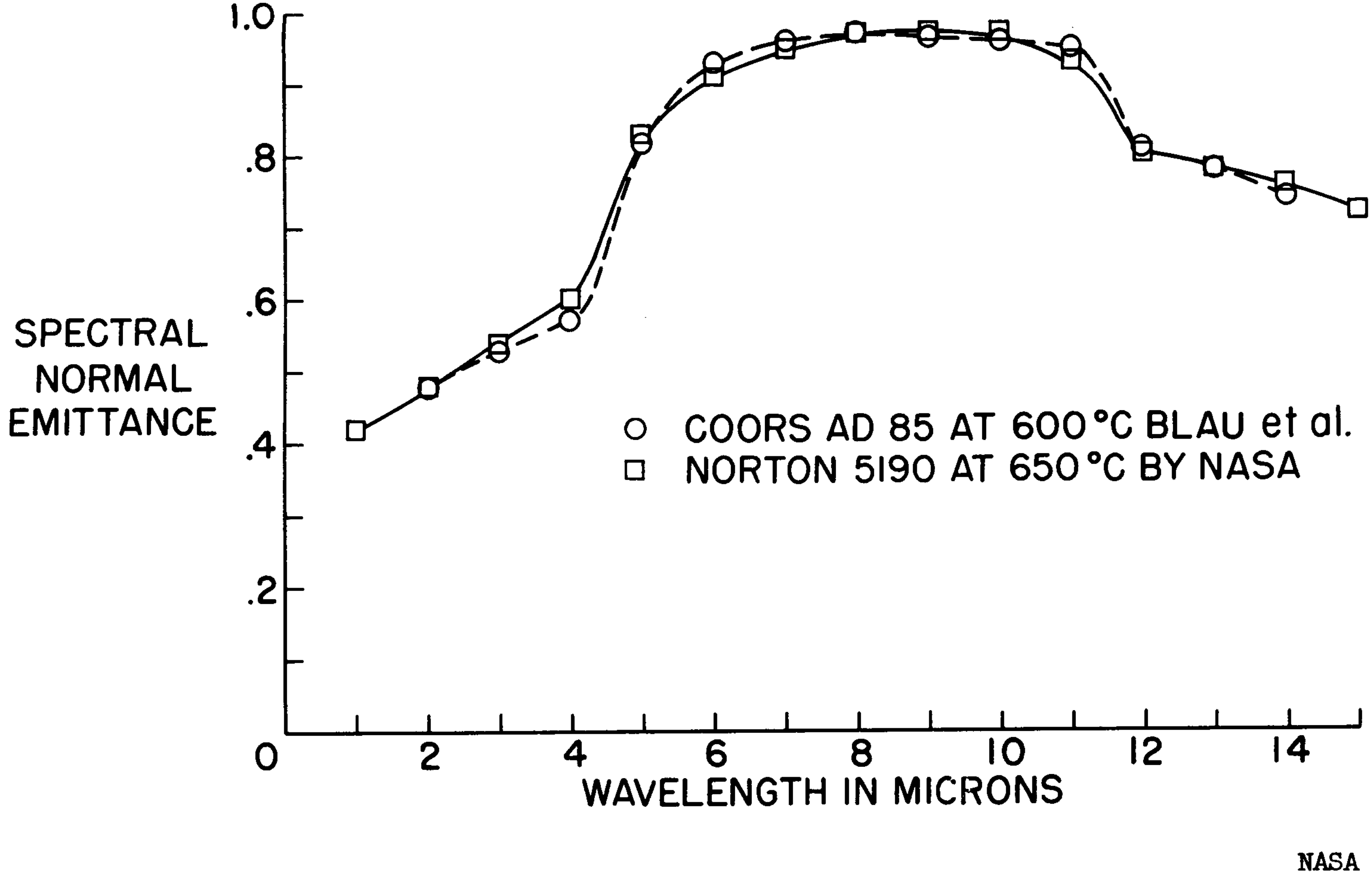

Figure 9.- Alumina.